APS Workshop 5: Integrating Nanofabrication with Next Generation X-ray Techniques to Probe and Control Novel Phenomena

Thursday, May 9, Morning

8:30 – 8:40 Gilberto Fabbris (Argonne National Laboratory)
Workshop Introduction

8:40 – 9:05 David Czaplewski (Argonne National Laboratory)
CNM Capabilities and Review of APS Collaborations

9:05 – 9:30 Alexander High (University of Chicago)
Engineering Quantum Properties in Diamond with Strain

9:30 – 9:55 Stephan Hruszkewycz (Argonne National Laboratory)
In-situ *X-ray Characterization of SiC Polytypic Heterostructure Synthesis*

9:55 – 10:10 Preetha Sarkar (University of Illinois at Urbana-Champaign)
Strain Superlattice Effects on the Electronic Properties of Graphene

10:10 – 10:30 Break

10:30 – 10:55 Claire Zurkowski (Lawrence Livermore National Laboratory)
Achieving Quasi-hydrostatic Equations of State in the Toroidal DAC: The Case Study of Molybdenum

10:55 – 11:20 Pedro Lozano (Argonne National Laboratory)
Nanofabrication of Extreme Sample Environments

11:20 – 11:45 James Walsh (University of Massachusetts Amherst)
Stoichiometric Control in the Diamond Anvil Cell Using Magnetron Co-sputtered Precursors

11:45 – 12:00 Eduardo Poldi (University of Illinois at Chicago)
Nitrogen-incorporated Ultrananocrystalline Diamond Electrodes on Single-crystal Diamond Anvils

12:00 Adjourn

Friday, May 10, Morning

8:30 – 8:55 Zachary Geballe (Carnegie Institution of Washington)
Diamond Anvil Micro-assemblies for Exploding Wire Experiments and Other Geometrically Demanding Experiments at Megabar Pressures

8:55 – 9:20 Luiz G. Pimenta Martins (Harvard University)
Nano- and Microfabrication Techniques to Load Atomic-thick Materials into Diamond Anvil Cells

9:20 – 9:45 Stanley Tozer (Florida State University)
The Marriage of FIBaro: Focused Ion Beam Enabled Mbar Superhydride Studies

9:45 – 10:00 Zackary Rehfuss (Washington University)
Electron Beam Patterning of Diamond Culets for High Pressure Transport Experiments

10:00 – 10:30 Break

10:30 – 10:55 Jeffrey S. Pigott (Case Western Reserve University)
Nanofabrication of Samples for Laser-heated Diamond-anvil Cell Experiments

10:55 – 11:20 Shua Sanchez (Massachusetts Institute of Technology)
Novel Device for Strain-tuning 2D Materials

11:20 – 11:45 Jiarui Li (Stanford University)
Strain Control of Electronic Orders in Oxide Nano Membranes

11:45 – 12:00 Discussion and Closeout

12:00 Adjourn

CNM Capabilities and Review of APS Collaborations

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The Center for Nanoscale Materials (CNM) at Argonne National Laboratory is a DOE Office of Science user facility, collocated with the APS, providing researchers from across the globe with world-class expertise and instrumentation for multidisciplinary nanoscience and nanotechnology research in topics ranging from biology to condensed matter physics to optics to quantum systems. Academic, industrial, and international researchers can access the CNM through our user program for both nonproprietary and proprietary research. Access is provided at no cost to users for research that is in the public domain and intended for publication. One of the key capabilities at the CNM is the large cleanroom equipped with a wide variety of state-of-the-art tools for lithography, deposition, etching, and metrology. Equally as valuable is the over 100 years of fabrication, process development, and process integration experience the CNM staff offer in support of the user program. In this talk, I will present information on these capabilities and select a few collaborative efforts between the CNM staff and the APS staff and users as examples of how collaborations can lead to excellent science.

Engineering Quantum Properties in Diamond with Strain

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We will discuss the interplay of strain and qubit performance in diamond membranes. Robust spin-photon interfaces in solids are essential components in quantum networking and sensing technologies. Group-IV color centers in diamond, such as the tin-vacancy center (SnV), are promising spin-photon interface with desirable optical and spin properties at 1.7 K. However, the SnV spin lacks efficient microwave control, and its spin coherence degrades with higher temperature. Here, we demonstrate that controllable strain generation in diamond membranes overcomes these challenges [1]. The controlled generation of crystal strain introduces orbital mixing that allows microwave control of the spin state with 99.36(9)% gate fidelity and spin coherence protection beyond a millisecond. Moreover, the presence of crystal strain suppresses temperature-dependent dephasing processes, leading to a considerable improvement of the coherence time up to 223(10) μs at 4 K, a widely accessible temperature in common cryogenic systems. Critically, the coherence of optical transitions is unaffected by the elevated temperature, exhibiting nearly lifetime-limited optical linewidths. Combined with the compatibility of diamond membranes with device integration, the demonstrated platform is an ideal spin-photon interface for future quantum technologies.

This work on strain engineering of group IV color centers is supported by the Air Force Office of Scientific Research under Grant No. FA9550-22-1-0518. This work acknowledges funding through Q-NEXT, supported by the U.S. Department of Energy, Office of Science, National Quantum Information Science Research Centers. Diamond growth related efforts were supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Materials Science and Engineering Division (N. D.)

[1] X. Guo*, A.M. Stramma*, Z. Li, W.G. Roth, B. Huang, Y. Jin, R.A. Parker, J.A. Martinez, N. Shofer, C.P. Michaels, C.P. Purser, M.H. Appel, E.M. Alexeev, T. Liu, A.C. Ferrari, D.D. Awschalom, N. Delegan, B. Pingault, G. Galli, F.J. Heremans, M. Atature† & A.A. High†, "Microwave-based quantum control and coherence protection of tin-vacancy spin qubits in a strain-tuned diamond membrane heterostructure," Phys. Rev. X 13, 041037 (2023).

In-situ X-ray Characterization of SiC Polytypic Heterostructure Synthesis

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SiC is a wide bandgap semiconductor with myriad applications, ranging from existing high-power electronics (e.g., power conversion in electric vehicles and grid components) to future high frequency and quantum communications and quantum sensing. Across these applications, developing synthesis protocols that yield suitable crystal quality and desired phase is critical and is complicated by the very high temperature synthesis window (1500-2000 C) of SiC and by the fact its many polymorphic phases. In the context of quantum information science, the high sensitivity of point defects ("color centers") to the local lattice structure, paired with the reported occurrence of SiC in over 200 polytypes (crystal structures with variations in the Si-C stacking sequence), creates an opportunity for versatile quantum sensing and communications technologies based on SiC. However, the close formation energies of the most common SiC polytypes, 3C-SiC, 4H-SiC, and 6H-SiC, often leads to the undesired synthesis of mixed polytype inclusions in nominally monopolytypic materials, which generates quantum wells that influence point defects in unintended ways. As a result, heteropolytypic inclusions and other lattice defects that alter color center properties (e.g., stacking faults) are typically thought of as detrimental. We propose instead to harness the additional tunability of color centers afforded by the intentional synthesis of SiC polytypic heterostructures, which could lead to new capabilities for the coherent control of SiC quantum defects as well as for nanofabrication and other device technologies.

Achieving reproducible, high quality SiC polytypic heterostructures requires a detailed understanding beyond what is currently known of the surface energetics and structural modifications during SiC growth that drive polytype stability. To this end, we have built an x-ray compatible *in-situ* chemical vapor deposition (CVD) chamber for SiC synthesis designed for use with hard x-rays (photon energy > 20 keV) that can penetrate the thick chamber walls of the CVD chamber to directly probe the SiC surface structure. This enables crystal truncation rod scattering measurements which are highly surface sensitive to different SiC polytypes as well as the terminal layer of the SiC stacking sequence. With the coming high brightenss of the APS-Upgrade, surface coherent diffraction studies can also be envisioned with x-ray photon correlation spectroscopy to directly observe growth modes as they relate to homo/hetero-epitaxy in SiC. We will share results from preliminary pre-upgrade results with this that pave the way for future work at the CHEX beamline. Such *in-situ* studies, when combined with modeling efforts that can reveal surface energetics such as density functional theory, can shed light on the fundamental connections between the parameters of the CVD synthesis environment, the material ultimately created, and the pathways to synthesize targeted polymorphs of high crystal quality.

Strain Superlattice Effects on the Electronic Properties of Graphene

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Strain superlattices in two-dimensional van der Waals materials have been predicted to lead to novel quantum phenomena, such as, generation of pseudo-magnetic fields and creation of flat electronic bands, which can give rise to zero-field Hall effect, valley Hall effects, topological edge states, and strongly correlated states of matter like superconductivity. However, there have been few experimental realizations of custom, reproducible superlattice potentials in 2D materials where primarily strain effects dominate. In this talk, I will discuss the techniques developed by Mason group for generating periodic strain potentials in graphene, by stacking it on nano-templated substrates, such as lithographically patterned nano-pyramids [1], nanopillars [2], and nanosphere self-assemblies [3]. Low temperature transport experiments provide key insight into how strain effects can modify the electronic band structure of 2D materials. In the second part of this talk, I will discuss the transport signatures that help us confirm the existence of a graphene strain superlattice [4,5] in the graphene-nanosphere (NS) systems, and how the strain induced pseudo-magnetic field can lift the valley degeneracy and lead to valley polarized edge currents. Scanning tunneling measurements on these graphene-NS devices help us confirm the existence of a spatially varying pseudomagnetic field as high as 55 T and the emergence of fractional pseudo-Landau levels [6].

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Achieving Quasi-hydrostatic Equations of State in the Toroidal DAC: The Case Study of Molybdenum

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The unit-cell volumes of molybdenum were tracked in the toroidal diamond anvil cell (tDAC) to 336(1) GPa at room temperature. To improve the compression environment in the tDAC, sample configurations were developed with molybdenum fully encased in copper. Copper was chosen, as it is a soft metal that does not withstand significant uniaxial stress with a well calibrated equation of state. Molybdenum was also compressed in neon between beveled anvils for comparison. The (200) lattice plane of Mo produced the lowest volumes across the pressure range of this study for all compression environments, indicating that this plane is least affected by uniaxial stress in the DAC. An equation of state of molybdenum based on the indexed (200) volumes and calibrated to copper pressure is presented for the copper and neon compression environments and compared to theoretical predications and extrapolations of Mo compressed in He.[1] Our EoS fits to the data collected in the tDAC agree with extrapolations of hydrostatic EoS fits within 1 % at 4 Mbar [1], while previous nonhydrostatic compression studies of Mo to 4 Mbar exhibit pressure differences of up to 13 % [2]. Results from this work demonstrate that utilizing copper to encase high-strength materials like molybdenum in the toroidal diamond anvil cell drastically improves sample hydrostaticity and results in measured sample volumes comparable to that collected in noble-gas media even at multi-megabar conditions.

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Nanofabrication of Extreme Sample Environments

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Applying external pressure in the study of materials allows one to control their electronic and magnetic orders, often resulting in novel emerging phenomena. A path to ultra-high static pressure generation has been opened by the introduction of toroidal diamond anvils. Owing to their reduced culet size and toroidal groove, these anvils have been shown to exceed static pressures of 600 GPa. In this talk, we will present the recent developments in the fabrication of toroidal anvils. The versatile, user-friendly fabrication process will be described in detail. We discuss the reproducibility of the method, as well as preliminary results. Finally, we highlight some of the new possibilities for users coming from combining toroidal anvils with the upgraded Advanced Photon Source.

Stoichiometric Control in the Diamond Anvil Cell Using Magnetron Co-sputtered Precursors

Paul V. Marshall[1], Scott D. Thiel[1], Elizabeth E. Cote[1], John Arigbede[1], Tracey Nelson[1], Dean Smith[2], Yue Meng[2], Rostislav Hrubiak[2], Matthew L. Whitaker[3], and James P. S. Walsh[1]

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Extreme pressure provides synthetic access to new materials that could propel next-generation or as-yet unimagined technologies. However, chemical control in the diamond anvil cell is difficult to achieve compared to traditional solid-state methods. In this talk, I will describe some new methods being developed in our lab that empower synthetic chemists with the tools they need to target and recover novel high-pressure phases, even within systems containing multiple competing phases on the convex hull. I will share some of our recent results on the discovery of novel transition metal carbides, demonstrating how our methods can allow the highly selective synthesis of high-pressure phases [1,2].

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Nitrogen-incorporated Ultrananocrystalline Diamond Electrodes on Single-crystal Diamond Anvils

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Recently some remarkable achievements regarding the stabilization of near-room-temperature superconductors [1,2] were only possible due to the use of diamond anvil cells (DACs). One of the critical aspects in the experiments that lead to such discoveries is to maintain electrical access to the sample in the DAC while applying pressure. In this work we present progress on the development of a unique type of designer diamond anvil cell for high pressure transport measurements [3]. Using a microwave plasma chemical vapor deposition process, we fabricated N-incorporated ultrananocrystalline diamond (N-UNCD) electrodes optimized for high pressure resistivity experiments. These electrodes were then covered with an UNCD insulating layer to prevent shorting with the metallic gasket that surrounds the sample. Our first tests were performed on a 300 μm-culet diamond anvil, but the procedures developed can be easily adapted for reaching higher pressures with diamonds of smaller culets.

N-UNCD presents resistivity of about 4 orders of magnitude higher than metals commonly employed in conventional high-pressure transport measurements like Pt or Au, but diamond electrodes provide better robustness, with deformations and failures occurring less frequently. Additionally, replacing metallic contacts with low Z, carbon-based probes decreases the interaction between sample and the instrument itself during DAC growth and characterization experiments. Furthermore, the four-point probing method for resistivity measurements tend to mitigate the overall effect of high-resistance electrodes, making all-diamond designer anvil cells an ideal instrument for reliable transport experiments, that can be combined with concomitant diffraction or spectroscopy techniques.

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Diamond Anvil Micro-assemblies for Exploding Wire Experiments and Other Geometrically Demanding Experiments at Megabar Pressures

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A well-prepared diamond anvil cell is crucial for many cutting-edge high-pressure experiments. For example, we have used pulsed Joule heating – also known as "exploding wire experiments" – to measure melting and resistivity of metals up to megabar pressures in diamond anvil cells. These experiments require a ~10-micron sample surrounded by many additional components such as electrodes and thermal insulation. Crucially, the components must be arranged in such a way that they end up with appropriate shape and location within ~1- to-10-micron tolerance after a large amount of compression, and inevitably after large shear strain. In this talk, I will present a new method of assembling a sample chamber for pulsed Joule heating DAC experiments by stacking three-layers, each of which has been pre-compressed with a subset of the necessary components [1]. The method seems to enable ~ 3-micron precision in sample dimensions up to 150 GPa and 4000 K. I will also discuss possible pathways for making the method less time-consuming and more accessible, and how we might use it to overcome long standing challenges in high pressure science.

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Nano- and Micro-fabrication Techniques to Load Atomic-thick Materials into Diamond Anvil Cells

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Two-dimensional (2D) materials and moiré superlattices formed by certain stacking configurations of 2D crystals, represent a new frontier in condensed matter research due the emergent properties associated to their reduced dimensionality and tunability. To glean insight into the physics of these atomically-thin van der Waals materials, their properties have been extensively studied by tuning of external parameters such as temperature, magnetic field, electrostatic doping, and strain. However, there is an external tuning parameter that has not been used systematically in studies of these systems – pressure [1]. The relative scarcity of high-pressure studies involving atomically-thin materials is due to experimental challenges, with a major one being loading atomically-thin micron-sized samples into the also micron-sized pressure chamber. In this talk, I will discuss different micro- and nano-fabrication methods developed by colleagues and me, to address the issue of loading 2D materials into diamond anvil cells (DACs). I will focus on two techniques: (i) a new sample fabrication method involving etching structures through 25-microns-thick silicon chips, which were used as substrates to consistently load 2D materials into DACs, and (ii) directly transferring 2D flakes sitting on appropriate nanometer-thick substrates such as graphite, onto the diamond's culet. I will briefly describe some high pressure optical spectroscopic studies that were carried out using those methods [2-4], with an emphasis on the experimental results that were only possible due to the choice of substrate. I will conclude with possible directions to integrate those techniques for the fabrication of atomic- and nanometer-thick samples for high-pressure optical spectroscopy and x-ray research.

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The Marriage of FIBaro: Focused Ion Beam Enabled Mbar Superhydride Studies

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In a cycle that has repeated itself for several decades, new discoveries just out of reach of then present-day high-pressure capabilities have driven each other forward. To wit: after years of unsuccessful attempts to create metallic hydrogen (a potential room temperature superconductor) by direct compression of the gas, Ashcroft [1] proposed that chemical pre-compression of hydrogen in a host metal lattice might be a more feasible route to this goal. Computationalists [2] led the effort to identify binary candidates, and a 2015 prediction of superconductivity in H_3S with $T_c > 200$ K [3] was simultaneously realized by Eremets [4]. A recent independent verification [5] of this result clearly shows a less than ideal electrical pathway which leads to a non-zero resistance yet confirms the initial discovery. Prior to this confirmation, a flurry of activity drove the transition temperatures in the binary superhydrides to 250-270 K in LaH_{10} [6,7]. Subsequently, we synthesized a higher order La-based superhydride with initial superconducting transition temperature of 294 K that, when subjected to thermal cycling, morphed into a higher order system with an onset T_c of 556 K [8]. X-ray and electrical transport data for this sample (the setting for which has been deemed both ugly and crappy) support one another with regard to the pressure measured, materials present, and the inhomogeneous nature of the synthesis that resulted in a broad multiphase transition and a non-zero background below T_c. These less-than-ideal results are to be expected given the use of ammonia borane (AB) as the hydrogen source and pressure medium in most studies and the "crucible" for superhydride growths, typically a Ø25 µm x 2 µm deep hole in the cBN gasket insert. The FIBed electrical leads, that are embedded in this "crucible," probe a pathway through this inhomogeneous growth, a very small portion of which is the superconductor of interest with most of the contents being non- or off-stoichiometric compounds or unreacted host metal. Compare that to the more traditional ambient pressure growth in a large volume crucible that can be held at precise homogeneous temperatures for specified times and from which individual crystals are extracted and measured for optimal properties and it is clear that superhydride research is still very much in the discovery phase. FIBed electrodes on diamond anvils played a significant role in our discovery, and we are now using this same technique to pattern pickup coils and define sample geometry and devices in an effort to validate the result and shed light on the underlying physics.

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Electron Beam Patterning of Diamond Culets for High-pressure Transport Experiments

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In recent years, the manipulation and control of quantum phases have emerged as central themes in scientific inquiry. However, implementing such control within a diamond anvil cell has presented significant experimental hurdles. Here, we introduce a robust patterning technique for directly depositing microcircuits onto the diamond culet and facets. This method, employing a precise trilayer patterning process involving titanium, chromium, and gold deposition, facilitates high-pressure transport experiments on both bulk and thin flake samples. The process begins with a bilayer resist on the diamond encapsulated by a thin aluminum layer, followed by patterning using electron beam lithography. Sequential steps involve etching with aluminum etchant and electron beam deposition to deposit the metal layers, concluding with a liftoff process to eliminate excess metal. This methodology offers a concrete pathway for investigating quantum phenomena under extreme conditions, promising significant advancements in our comprehension of quantum materials.

Nanofabrication of Samples for Laser-heated Diamond-anvil Cell Experiments

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Accurate and precise synchrotron-based experimental measurements of high-pressure, high-temperature equations of state, and phase diagrams are required for robust modeling of planetary interiors and shock phenomena. However, these experiments face obstacles such as large temperature gradients within the sample volume probed by the x-ray beam, unwanted chemical reactions, containment of material after melting, and slight optical misalignments. In this presentation, I will discuss microfabrication and nanofabrication methods that can be used to create samples tailored to laser-heated diamond-anvil cell experimental applications. I will highlight equation of state studies using nanofabricated Ni/SiO_2/Ni samples [1] and discuss progress in nanofabricating multi-layer samples of different metals and SiO_2 which prevents alloying of the metal layers. Finally, I will provide an overview of available facilities at Case Western Reserve University for nanofabrication and microfabrication and discuss methods for characterization of nanoparticles and thin film devices that could be leveraged for novel experiments enabled by the forthcoming upgraded capabilities at the Advanced Photon Source.

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Novel Device for Strain-tuning 2D Materials

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In this talk, I will introduce a new approach for strain-tuning 2D materials. We have used photolithography to create an etched silicon device with a very small (~5 um) gap, across which an ultrathin sample is mounted. By applying strain to the device via piezo actuators, the size of the gap can be tuned, which transmits strain to the sample. I will discuss using this device combined with x-ray diffraction and spectroscopy techniques to probe strain-tunable electronic orders in 2D materials.

Strain Control of Electronic Orders in Oxide Nano Membranes

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Quantum materials, especially in novel forms, allow precise control over material properties, driving advancements in future quantum technology. By synthesizing quantum materials in the membrane form, superior mechanical properties can be achieved that are otherwise unattainable in bulk forms. In this talk, I will present our recent progress in the integration of extreme tensile strain control in quantum material membranes with advanced x-ray techniques. I will highlight a case study in single crystal membranes of $SrTiO_3$, where a fully reversible quantum paraelectric to ferroelectric transition is induced via precise control over lattice parameters up to 1.5%. Temperature-strain phase diagram of emergent ferroelectric order has been constructed. Our study not only sheds light on the nature of the quantum paraelectric to ferroelectric transition in $SrTiO_3$ but also paves the way for a deeper comprehension of membrane-based quantum materials through multimodal synchrotron probes.